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ATTORNEY DOCKET NO.: P1038

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: Mercuri *et al.* :

SERIAL NO: 09/871,243 : ART UNIT: 1774

FILED: May 5, 2001 : EXAMINER: Nguyen, K.

FOR: Method for Preparing Composite Flexible Graphite Material :

Asst. Commissioner for Patents
Washington, D.C. 20231

I HEREBY CERTIFY THAT THIS CORRESPONDENCE IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE AS FIRST-CLASS MAIL IN AN ENVELOPE ADDRESSED TO: ASST. COMMISSIONER FOR PATENTS AND TRADEMARKS, WASHINGTON D.C. 20231 ON THIS 27th DAY OF January 2003
BY: Carrie A. McPherson

DECLARATION OF DR. JEREMY H. KLUG UNDER 37 C.F.R. 1.132

1. I, Jeremy H. Klug, am a named inventor in the above captioned matter.
2. In 1993 I earned a Bachelor of Science degree from the University of Minnesota in Chemical Engineering.
3. In 1999 I earned a Doctorate degree from the University of Washington in Chemical Engineering.
4. In 1999 I started working for the UCAR Carbon Company in areas of carbon and graphite science in their flexible graphite business as a staff scientist. In 2001, I was promoted to the position of research scientist for the same business for the aforementioned corporation and/or its successors in interest.
5. As part of my duties as research scientist, I organized and oversaw the preparation and the thru-plane testing of several combinations of flexible graphite sheets in accordance the elements of amended claim 1, a monolith flexible graphite sheet, and a flexible graphite composite which the basis weight of the flexible graphite sheets in the composite and the resin weight % in each sheet did not vary.
6. The samples evaluated as applied to the amended claim 1 are described in the below table 1.

Table 1

Sample ID	Description of Sample
A	Monolith of flexible graphite with 33% resin by weight
B	2 sheets of flexible graphite each sheet had a basis weight of graphite of 60 mg/cm^2 and a resin weight percent of 10%
C	One flexible graphite sheet had a basis weight of graphite of 70 mg/cm^2 and a resin weight percent of 33% and second flexible graphite sheet had a basis weight of graphite of 8 mg/cm^2 and a resin weight percent of 0%
D	One flexible graphite sheet had a basis weight of graphite of 60 mg/cm^2 and a resin weight percent of 10% and second graphite sheet had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%
E	One flexible graphite sheet had a basis weight of graphite of 60 mg/cm^2 and a resin weight percent of 10% and second graphite sheet had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%
F	Bottom 4 flexible graphite sheets had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0%, 5th flexible graphite sheet had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%, and top flexible graphite sheet had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0%
G	1st, and 5th sheets of flexible graphite had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0%, 2nd and 4th flexible graphite sheets had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%, and 3rd sheet of flexible graphite had a basis weight of graphite of 8 mg/cm^2 and a resin weight percent of 0%
H	1st, 3rd, and 5th sheets of flexible graphite had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0% and 2nd and 4th flexible graphite sheets had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%
I	The outside sheets of flexible graphite had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0% and the two interior sheets of flexible graphite had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of

	27%
J	The 1st sheet of flexible graphite had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0%, 3rd and 5th sheets of flexible graphite had a basis weight of graphite of 8 mg/cm^2 and a resin weight percent of 0%, and 2nd and 4th flexible graphite sheets had a basis weight of graphite of 30 mg/cm^2 and a resin weight percent of 27%
K	1st, 3rd, and 4th flexible graphite sheets had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0% and 2nd flexible graphite sheet had a basis weight of graphite of 60 mg/cm^2 and a resin weight percent of 10%
L	1st, 3rd, 4th, and 5th flexible graphite sheets had a basis weight of graphite of 16 mg/cm^2 and a resin weight percent of 0% and 2nd flexible graphite sheet had a basis weight of graphite of 60 mg/cm^2 and a resin weight percent of 10%

7. The through-plane electrical resistance of each sample was tested using an electrode pressure of 200 psi and a current density of 1 A/cm^2 . The resulting voltage drop values of such testing are provided in the below table 2. All sample thickness values were within +-0.06 mm of the nominal value of 0.95 mm.

Table 2

Sample ID	Thru-Plane Resistivity (mV) After cure
A	16.4 +-0.7
B	15.9 +-0.7
C	8.5 +-0.4
D	15.8 +-2.1
E	12.4 +-0.2
F	6.0 +-0.2
G	6.5 +-0.1
H	6.6 +-0.1
I	6.2 +-0.1
J	6.6 +-0.1

K	5.9 +-0.1
L	6.5 +-0.2

8. The samples which had a difference in either basis weight or resin content of greater than 5% demonstrated a resistance of less than both the monolith sample (A) and the sample without a 5% difference in a characteristic (B). In nine of the ten samples, the reduction in resistance was greater than 10% and in 7 of the 10 samples, the resistance was less than 50% of samples A and B.

9. In conducting the experiment, I did not expect the differences in resistance exhibited by samples C-L as compared to the resistance exhibited by samples A and B.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dr. Jeremy H. Klug

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Fiber Reinforced Plastics

In fiber reinforced plastic applications with fire safety concerns or high-temperature requirements, phenolic resin systems have the clear advantage over polyesters, epoxies, and vinyl esters. Cured phenolic resins are inherently fire-resistant, demonstrate low smoke generation and high-temperature resistance, and supply superior strength. We offer a complete line of phenolic resins for the following processes that serve the aerospace, construction, mass transit, mining, marine, and offshore industries:

INTERNATIONAL DISTRIBUTOR

Our FRP resins are also available in Europe through our UK distributor Aeropia.

Pultrusion

We offer phenolic resins that can process on polyester pultrusion systems. The resins are styrene-free and are thermally cured without the need for external catalysts. Market applications include grating for offshore platforms.

Sheet and Bulk Molding Compounds (SMC/BMC)

We have developed a phenolic resin system for SMC and BMC that demonstrates long-term durability and resistance to hydrocarbon and chlorinated solvents. The viscosity, processing and cure properties of our phenolic system match those of polyester resins, thus avoiding the need for any special processing or equipment adjustments.

Hand Lay-Up and Resin Transfer Molding (RTM)"sin Transfer Molding (RTM)

Several phenolic resins and acid catalysts, as well as a phenolic-based in-mold surface paste, are available for the hand lay-up/RTM process. Phenolic technology has advanced to achieve polyester-like processing, mechanical properties, pot life and cure speed. The phenolic resin and paste systems have both passed United States, British, French, and International Maritime Organization Standard Fire Tests.

Honeyc mb and Prepreg

We offer phenolic resins for Nomex honeycomb, glass fiber prepregs and carbon fiber prepregs for aerospace

applications. The product line includes solventborne and waterborne resins that meet Military Specification MIL-R-9299C requirements.

Filament Winding

We have developed phenolic resins with latent acid catalysts to meet filament winding processing requirements. Applications include mine ventilation, air conditioning systems, clean room ducting, and offshore water hydrant pipe systems.

FRP composites are used to replace typical traditional structural materials of steel, metal, aluminum and wood. Composites offer numerous advantages over traditional materials - low density, lighter weight, corrosion resistance, high strength, good impact resistance, excellent electrical properties, and durability.

REMR MATERIAL DATA SHEET CM-CR-1.5

EPOXY RESIN SYSTEM FOR DORMANT CRACK REPAIR AND SURFACE SEALER: DELTA AS69-9046 UNDERWATER BONDING ADHESIVE

1. NAME

Delta AS69-9046
Underwater Bonding Adhesive

2. MANUFACTURER

Delta Plastics Company
7449 Avenue 301
Visalia, California 93291
Telephone: 209-851-2034

3. DESCRIPTION

Delta AS69-9046 A&B System is a medium viscosity, 1:1 mixing ratio by volume, medium pot life and medium gel time material developed specifically for use in making underwater repairs of masonry materials. The resin-hardener mixture will cure under fresh or salt water and will bond to clean wet surfaces. This system cures to a rigid tough adhesive with excellent bond strengths to masonry surfaces and to many other types of material surfaces including polystyrene foam. Other unique features of this system are that it will cure at low temperatures (40°F) and the cured system has excellent chemical resistance.

4. APPLICABLE SPECIFICATION

Specification of ASTM C 881-78, "Standard Specification for Epoxy-Resin-Base Bonding Systems for Concrete," generally apply.

5. USES AND LIMITATIONS

Uses: This system is suggested for use in underwater applications for the repair of cracks in masonry materials, the adhesive bonding of concrete, plastics, metals, ceramics, etc., to masonry surfaces, and the coating of the same type of materials underwater. This system has been successfully used to repair cracked concrete underwater. The material was pumped, under pressure, by in-head mixing equipment and injected through a nipple located at the bottom of the crack. The water in the crack was displaced as the epoxy adhesive filled the crack.

Limitations: A guide to the gel times and cure times at various temperatures for a 200 gram mass is as follows:

Temperature	Gel Time	Full Cure Time
50°F	33 to 40 min	7 days
70°F	17 to 23 min	5 days

Mix only the amount of material at one time that can be used within the times and temperatures given. Less mixed material will increase the times shown, while larger batches will decrease the times shown. The addition of sand or other similar types of aggregate to the resin-hardener mixture will also increase the times shown.

6. MANUFACTURER'S TECHNICAL DATA

Property	Value
Part A, c lor	Slight amber
Part B, color	Slight amber
Mixed, color	Slight amber
Solids content, percent	100
Mixed viscosity, cps	4,000
Gel time, 200 gm mass, 70°F	20 min
Hardness, short	D-68
Tensile strength, psi	7,800
Tensile elongation, percent	12
Compressive strength, psi	16,900
Fl xural strength, psi	15,800
Flexural modulus of elasticity, psi (1×10^6)	0.51
Coefficient of linear thermal expansion (in./in./C)	54×10^{-6}

Slant shear bond strengths (cure 7 days @ 77°F)

Material	Cured Under Water	Cured in Air
Concrete to concrete*	Concrete fractures	Concrete fractures
Steel to concrete*	Concrete fractures	Concrete fractures
Steel to steel**	2,450	2,900
Cr osoted wood to creosoted wood**	Wood fractures	Wood fractures
Aluminum to aluminum**	2,200	2,600

* Direct shear
** ASTM D 1002

7. MANUFACTURER'S GUIDANCE FOR APPLICATION

Surface Preparation:

Th surfaces to be bonded must be clean. Plant or animal life, slime, oxides, laitance, etc., must be removed. Grit blasting or ultra high pressure water blasting may be the only effective methods of cleaning some structures.

Mixture Proportioning: The ratio of resin and hardener must be accurate.

Part A	95 parts by weight or 1 part by volume

Part B	80 parts by weight or 1 part by v lume

The adhesive components ar medium-viscosity materials, and thorough mixing in air before using is essential. Ke p air entrapment to a minimum.

Application: After mixing the adhesive thoroughly in air, make the repair underwater.

The curing process starts when the adhesive is mixed. Low temperatures lengthen the gel time and thus increase the risk that the adhesive may wash away. Therefore, in cold weather and in flowing water, it may be helpful t apply the mixed adhesive to a backing material such as fiberglass, polyethylene, etc., prior to g ing underwater. The repair can then be made by applying the adhesive-coated backing material to the area.

Precautions: Always wear protective clothing and practice cleanliness when working with epoxy resins and hardeners. If skin contact is made, wash the area with soap and water. If the materials get into the eyes, rinse with quantities of water and consult a physician. Use epoxy resins and hardeners only in well-ventilated areas, and avoid breathing vapors.

8. CORPS OF ENGINEERS' EVALUATION

Technical data:

Performance Properties at 73°F	Test Method	Results
Viscosity, cp	ASTM D 2393	3,950
Gel time, min	ASTM C 881	27.5
N nvolatile content, percent	ASTM D 1259	96.5
Bond to concrete, psi	ASTM C 882	4,170 concrete failure
Effect of moisture on bond strength, psi	ASTM C 882	4,080 concrete failure
Compressive strength, psi	ASTM D 695	16,100
Young's modulus of elasticity, psi	ASTM D 695	4.23×10^5
T nsile strength, psi	ASTM D 638	7,890
Tensile elongation, percent	ASTM D 638	11.3
Flexural strength, psi	ASTM D 790	15,100
Shrinkage volumetric, percent		3.8
Hardness	ASTM D 2240	80
Water absorption, percent	ASTM D 570	0.33

9. ENVIRONMENTAL CONSIDERATIONS

Reasonable cauti n sh uld guide the preparation, repair, and cleanup phases of activities involving potentially hazardous and toxic chemical substances. Manufacturer's recommendations to protect occupational health and nvironmental quality should be carefully followed. Material safety data sheets should be obtained from the manufacturers of such materials. In cases where the effects of a chemical substance on occupational health or environmental quality are unknown, chemical substances should be treated as potentially hazardous toxic materials.

10. AVAILABILITY AND COSTS

Availability: The Delta AS69-9046 A&B System and the AS69-46 System are available in the following standard kit sizes packaged by weight:

	10-Gal Kit	2-Gal Kit	2-Qt Kit	1-Qt Kit
P rt A, weight	47.5 lb	8.5 lb	2.375 lb	1.2 lb
	10-Gal Kit	2-Gal Kit	2-Qt Kit	1-Qt Kit
Part B, weight	40 lb	8 lb	2 lb	1 lb

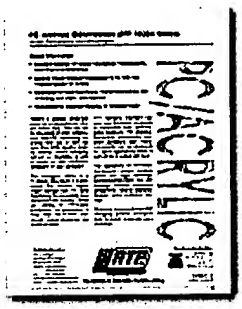
The material can be purchased at distributors throughout the U.S.A.

Costs: Delta AS69-9046 costs approximately $100 per 2-gal kit.

11. TECHNICAL SERVICES

The Delta Plastics Company facilities and help are available for specific problems.

PC/Acrylic Alloy Compounds



The RTP 1800A Series (PC/Acrylic Alloy) offers greater impact strength than PC and maintains the ease of processing associated with acrylic materials. These compounds can be processed at lower temperatures than PC, decreasing the cooling time and cycle time of the injection molding process.

While the PC/Acrylic resin system is already available in the marketplace, the RTP 1800A Series opens new application niches by incorporating wear additives, flame retardants, EMI shielding and PermaStat® permanently anti-static protection. The compound's natural opaque white provides excellent base for a wide range of colors. Compounds can also be developed for use in applications where a part will be in contact with food.

For more information on PC/Acrylic Alloy compounds, contact RTP Company at (800) 433-4787 or e-mail rtp@rtpcompany.com.

PC/Acrylic File Download

A version of the PC/Acrylic Compounds innovation bulletin suitable for printing or saving is available as a PDF (Portable Document Format) file. PDF files require the Acrobat Reader software for viewing.

More PC/Acrylic information

- RTP Company earns UL recognition for PC/acrylic and polytrimethylene terephthalate (PTT) compounds
- RTP Company announces new PC/Acrylic alloy compounds
- Resin system overview
- Product data sheet for compounds using PC/Acrylic alloy

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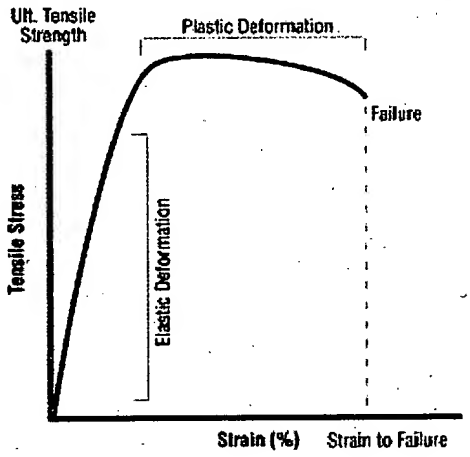
Resin Systems

Any resin system for use in a composite material will require the following properties:

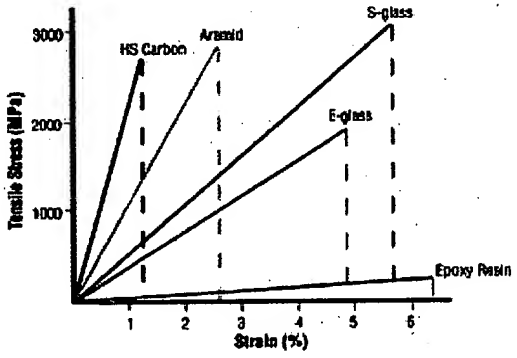
1. Good mechanical properties
2. Good adhesive properties
3. Good toughness properties
4. Good resistance to environmental degradation

Mechanical Properties of the Resin System

The figure below shows the stress / strain curve for an 'ideal' resin system. The curve for this resin shows high ultimate strength, high stiffness (indicated by the initial gradient) and a high strain to failure. This means that the resin is initially stiff but at the same time will not suffer from brittle failure.



It should also be noted that when a composite is loaded in tension, for the full mechanical properties of the fibre component to be achieved, the resin must be able to deform to at least the same extent as the fibre. The figure below gives the strain to failure for E-glass, S-glass, aramid and high-strength grade carbon fibres on their own (i.e. not in a composite form). Here it can be seen that, for example, the S-glass fibre, with an elongation to break of 5.3%, will require a resin with an elongation to break of at least this value to achieve maximum tensile properties.



Adhesive Properti s of th Resin System
High adhesion between resin and reinforcement fibres is necessary for any resin system. This will ensur that th loads are transferred fficiently and will prevent cracking or fibre / resin debonding when stress d.

Toughness Properti s of the Resin System
Toughness is a measur of a mat rial's r sistance to crack propagation, but in a composit this can be hard t measure accurately. How ver, the stress / strain curve of the resin system on its own provides some indication of the material's toughness. Generally the more deformation the resin will accept before failure the tougher and more crack-resistant the material will be. Conversely, a resin system with a low strain to failure will tend to create a brittle composite, which cracks easily. It is important to match this property to the elongation of the fibre reinforcement.

NEXT PAGE
PREVIOUS PA

Environmental Properties of the Resin System
Good resistance to the environment, water and other aggressive substances, together with an ability to withstand constant stress cycling, are properties essential to any resin system. These properties are particularly important for use in a marine environment.

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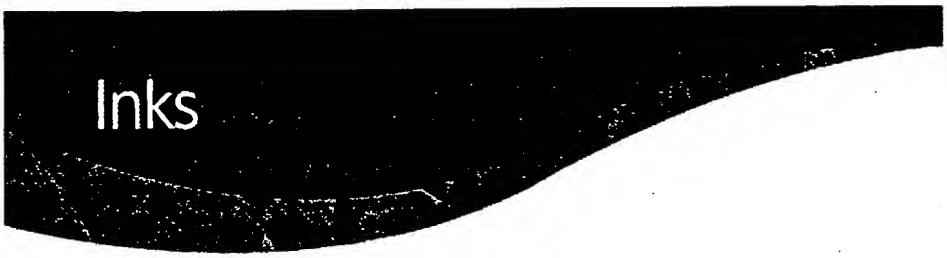

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rders

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Ink Resin Systems

Nitrocellulose/Polyurethane and Nitrocellulose/Polyamide Inks | Polyvinyl butyral-based (PVB) Inks

Nitrocellulose/Polyurethane and Nitrocellulose/Polyamide Inks

VERTEC IA10 and **VERTEC** PI2 are effective in a variety of resin systems which have nitrocellulose (NC) as the principal film former and usually, incorporate a co-binder such as polyamide or fully reacted polyurethane. They bring the following demonstrable benefits.

- Improved adhesion to OPP film
- Improved heat stability depending on the grade of OPP film used
- Reduced yellowing effects with **VERTEC** IA10 compared with titanium acetylacetonates
- No reduction in gloss with **VERTEC** IA10
- Improvement in the performance of acrylics, alkyd and maleic resins used with nitrocellulose

Polyvinyl butyral-based (PVB) Inks

Whilst adhesion of PVB based inks to polypropylene films is good, it can be improved further by the addition of **VERTEC** IA10 and **VERTEC** PI2. The effects of **VERTEC** products on heat stability depend on the ink formulation and substrate. Improvements can be achieved at up to 160°C. The addition of a stabiliser such a malic acid to control the ink viscosity is particularly important.

Synetix

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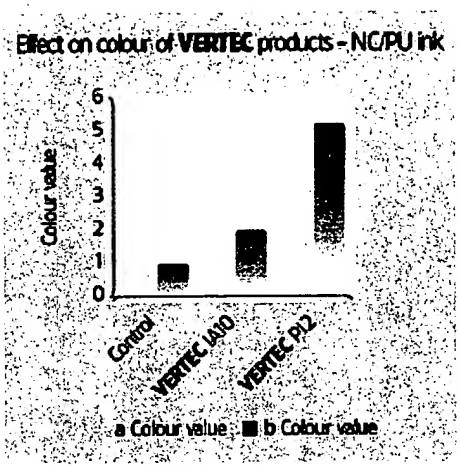
Effect on colour of VERTEC products - NC/PU ink
Colour value
6
5
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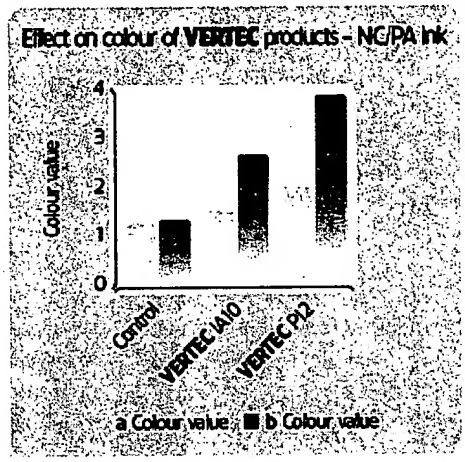
Control
VERTEC IA10
VERTEC PI2
a Colour value
b Colour value


Effect on colour of VERTEC products - NC/PA ink
Colour value
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Control
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VERTEC PI2
a Colour value
b Colour value

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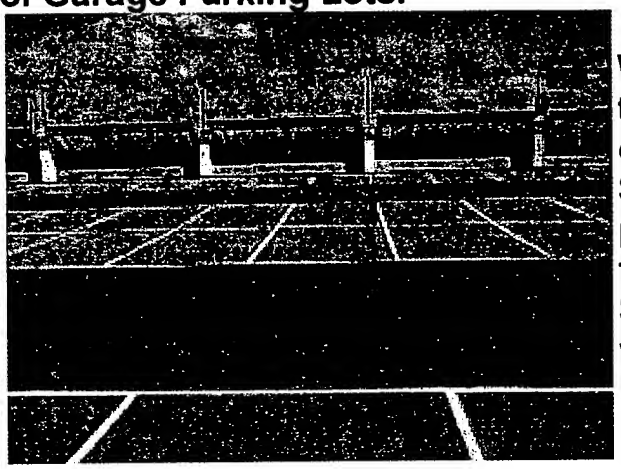
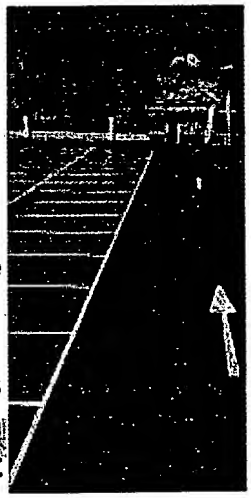


Waterproofing of parking to protect existing re-e concrete substrates by SRM Flex 1000 or polyurethane membrane fast installation and cur Systems cut revenue los works are carried out.



Re-surfacing of wearing courses and colour marking with SRM Grip 2000 and 4000 Polyurethane Resin Anti-Skid Systems. With fast installation and cure of the System cutting revenue lose while works are carried out.

Full lining service for exposed parking decks using SRM Spray Thermo Plastic Resin System. Works can be carried out in quiet periods to eliminate loss of revenue.

- Concrete repairs to existing parking decks, using th epoxy resin and fast cure repair mortars.
- Crash Barrier installation or replacement.
- Signing, barrier, and revenue collecting equipment, sup installation.
- Lighting and p rsonal safety surveys and installation of r equipm nt.
- Prot ctiv paint coatings for concr te and steel structur

Utilising SRM's Contact Management and fully trained Con Teams we can offer Turn Key Project Managem nt to fulf needs.

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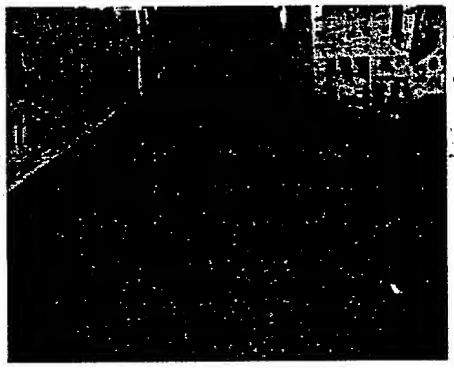
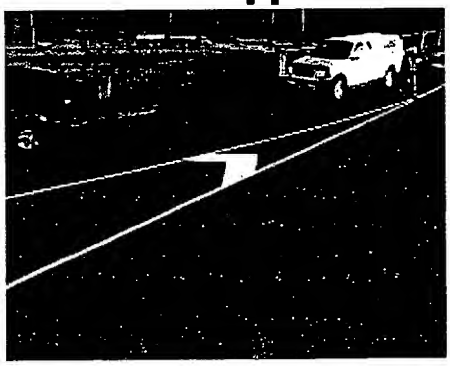


SRM are the North American for a range of World Polyurethane Resin Systems.

These Systems offer solutic many safety problems at the same time offering excellent corrosion control properties.

With applications in Marine, Highways, Bridges, Industry, a Parking, the Grip and Flex S can offer bespoke solutio particular problems.



Due to the rapid cure times of these products SRM's fully trained Contract Teams can install these systems with minimum revenue loss or disruption to client op rati

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